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(54) Tooth paste and gel compositions

(57) In these compositions for tooth pastes and gels, the exclusive or principal constituent adopted as a balanced cleanser-polisher, adsorbent and bleaching agent, is activated carbon, preferably of vegetable origin, granulated in fine particles e.g. of average diameter 2-20 microns with a surface area preferably of at least 300 m<sup>2</sup>/g. The activated carbon can be blended with suitable excipients at ambient temperature in a conventional mixer, and preferably comprises 0.5-50 wt% of the composition.

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## Description

TOOTH PASTE AND GEL COMPOSITIONS AND A METHOD  
FOR THEIR PREPARATION

The invention relates to new chemical compositions for tooth paste and gel products, and to a method for their preparation.

05 It is generally appreciated that the essential purpose of a toothpaste, in conjunction with a suitable brush, is that of ensuring an efficient and thorough cleansing action on the user's teeth. Brushing is designed fundamentally to bring about the removal of dental plaque, the gelatinous white  
10 deposit which forms at the edge of the gums, also of the film of proteinaceous origin which appears as a more or less visible stain, and of residual particles of food trapped between the interdental surfaces.

15 To ensure that these functions are carried out to best possible effect, the paste or gel product must be formulated to include cleansing and abrasive agents such as will remove of any manner of dirt or residue from all surfaces of the tooth, but without  
20 damaging the enamel and/or dentine, and/or the

delicate neighbouring gingival mucosa. In effect, where the substances utilized, be they natural or synthetic, are unsuitable in terms of structure, hardness, granulometry, uniformity or purity, this  
05 can lead to excessive scoring and/or abrasion of the enamel and/or dentine, and localized damage to tooth surfaces, which effectively are 'toothpaste scars'.

As demonstrable from pertinent prior art, numerous  
10 difficulties are encountered in formulating pastes and gels with the end in view of uniting all such properties as are necessary to an efficient oral hygiene in a single preparation; in particular, there is the need to remove dental plaque and thus  
15 avoid its negative consequences, to prevent any build-up, hence thickening and modification, of the protein film deposited on the enamel, and indeed to generate a preventive and complementary action in the treatment of pathological processes affecting  
20 the organs of the oral cavity such as are caused by a partial or total lack of oral hygiene, namely inflammation, bleeding, gingival atrophy, dental caries, halitosis, etc...

The formulation difficulties in question can be  
25 summarized briefly as a number of requirements:

- a) for an effective cleansing and polishing action over all dental surfaces, interstitial especially, without abrading or scoring the enamel;
- b) for a vigorous adsorbent and bleaching action;
- 05 c) for a uniform distribution of the constituents in the product, essential in order to ensure an action conducive to efficient oral hygiene;
- d) for stability of the paste or gel, and for a guarantee of its effectiveness especially in the
- 10 case of a composition featuring prophylactic or therapeutic properties.

Accordingly, the specific object of the invention is one of setting forth and preparing chemical compositions for tooth pastes and gels such as will

15 overcome or substantially reduce the difficulties described above.

The stated object is realized, with others besides, in paste and gel compositions and a method for their preparation according to the invention, as

20 characterized in the appended claims and described by way of example in the following specification.

The essential feature of the paste/gel compositions disclosed is that the exclusive, or the principal cleansing-polishing and adsorbent-bleaching agent,

25 is an orally innocuous activated carbon, finely

granulated and affording a generous developable surface area. Preferably, and given the obvious difficulties and costs associated with refining, the activated carbon will be of vegetable origin.

05 The chemical and physical properties of activated carbon in a multiplicity of different applications are well known, and require no detailed exposition; accordingly, the stated object consists essentially in exploiting these properties in the formulation  
10 of tooth pastes and gels.

According to the invention, between 0.5% and 50.0% by weight of the overall composition will consist in an orally innocuous activated carbon, that is, activated carbon of vegetable origin, prepared in  
15 granules of 2...20 m average diameter and having a developable surface area not less than 300 m<sup>2</sup>/g. Experiment has shown that the preferred particle granulometry is one reflecting an average diameter of 5...15 m and a developable surface area of  
20 between 600 and 800 m<sup>2</sup>/g.

A paste or gel composition according to the present invention comprises the conventional constituents embraced by the prior art, in quantities, needless to say, such as will give a cosmetically acceptable  
25 end product, typically: mono-, di- and tribasic

phosphates of alkaline and alkaline earth metals, metaphosphates, precipitated synthetic amorphous silicas, pyrogenics, silica xerogels, etc...

05 The new compositions may also contain excipients such as foaming agents (soaps and/or detergents of synthetic composition), sweeteners (saccharin and its salts, xylitol, glycerinated ammonium etc.), buffers, neutralizers, moisteners, preservatives, aromas, colouring agents, opacifiers (titanium  
10 dioxide) and polymeric lubricants and vehicles. The proposed compositions may also contain such quantities of vehicle-emollients, thickeners and binders as will ensure a homogeneous structure, the right viscosity, and a cosmetically acceptable  
15 appearance. Whilst preferred vehicle and emollient substances are glycerin and sorbitol, formulated either singly or in a suitable blend, diethylene, propylene or polypropylene glycols might also be utilized. Preferred binders and thickeners would  
20 include sodium carboxymethyl cellulose, hydromethyl cellulose and hydroxymethyl cellulose, though use might also be made of tragacanth, derivatives of starch, or certain synthetic silicas, for example. To advantage, the paste/gel compositions disclosed  
25 also contain decongestants and oedema-inhibiting

agents consisting in saline electrolytes such as sodium chloride and sodium bicarbonate, added at a rate of 2...60% by weight overall.

05 Finally, compositions according to the invention may incorporate prophylactic and/or therapeutic agents, such as astringents, water-soluble ionic compounds of fluoride, antibacterials, deodorants, decongestants, protective filmogens, and so forth. Typical examples are stannous fluoride, sodium  
10 monofluorophosphate, organic compounds of iodine, quaternary ammonium compounds, bisguanidines (e.g. 1,6-Di-N-p-chlorophenyl diguanido hexane), nitro-paraffin compounds (5-ammine-1,3-bis[2-ethylhexyl]-5-methylhexahydropyrimidine), natural and/or  
15 synthetic azulenes, 1-alpha-bisabolol, tannins, methylpolysiloxane, methylalkyl-polysiloxane, etc. In the compositions disclosed, such substances constitute between 0.01% and 5% by weight overall. The proportions for the various excipients will be,  
20 in general terms, vehicles and emollients 5...70%, binders 0.2...35%, aromas 0.1...5%, foaming agents 0.01...7%, buffers and neutralizers 0.01...3%, and water in such ever quantity is required to make up the remainder of the 100%; the percentages are  
25 expressed in terms of overall weight.

The invention also relates to a method for the preparation of the new activated carbon formulae disclosed, preferably in gel format; such a method comprises the following sequence of steps, brought about under a continuous, slow agitation:

05      a) preparation, at ambient temperature, either of a uniform dispersion of activated carbon in a medium of blended polyglycols, with sodium carboxymethyl cellulose admixed as a binder, or of a dispersion

10      of sodium carboxymethyl cellulose in polyglycols, blended or otherwise, and admixture thereto of the activated carbons;

15      b) admixture of deionized water to and heating of the uniform dispersion, preferably to a temperature of 50...60 °C;

20      c) admixture, in heat, of quantities of synthetic amorphous silicas sufficient to obtain an end product of cosmetically acceptable consistency, agitating until uniformity is achieved;

25      d) evacuation and deaeration;

         e) cooling the product to ambient temperature, and packaging in suitable containers using conventional techniques.

The basic method for the preparation of pastes or gels according to the invention, as outlined above,



has been researched and tested experimentally to the ends of obtaining the best and most uniform dispersion of the activated carbon, hence the best possible exploitation of its chemical and physical properties, and of establishing that the effects claimed for the product will indeed materialize in practice.

The method thus described might also comprise these further operations:

- admixture of sweeteners, buffers or neutralizers as part of step b);
- admixture of a saline electrolyte, in heat, prior to step c);
- admixture of foaming agents, between step c) and step d);
- admixture of prophylactic or therapeutic agents, and of aromatic substances, following the cooling operation of step e).

Utilizing an ordinary mixer for products of paste consistency, the method according to the present invention permits of preparing a uniform dispersion of the various constituents, and thus of obtaining a homogeneous paste or gel product of optimum and stable consistency, without any need for subsequent refining.

The following illustrations of possible gel and paste compositions according to the invention are given by way of example:

Composition - gel	% by weight
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Example 1: activated carbon as disclosed	10.0
glycerin	20.0
sodium saccharin	0.2
pyrogenic silica	10.0
buffer	0.9
germicidal	0.2
surface active compound	1.0
aroma	1.5
water - remainder	100.0
Example 2: activated carbon as disclosed	15.0
glycerin	20.0
silica thickening compound	9.0
sweetener	0.2
buffer	0.9
25%-solution sodium chloride	50.0
surface active compound	1.0
cetyl pyridinium chloride	0.2
aroma	1.0
water - remainder	100.0

Example 3:	activated carbon as disclosed	5.0
	glycerin	10.0
	70%-solution sorbitol	10.0
	sodium carboxymethyl cellulose	1.0
	pyrogenic silicas	6.0
	sodium chloride crystals	4.0
	25%-solution sodium chloride	20.0
	sweetener	0.2
	buffer	1.0
	sodium lauryl sulphate	1.0
	azulene	0.1
	aroma	1.5
	water - remainder	100.0

Example 4:	activated carbon as disclosed	15.0
	glycerin	20.0
	binder	5.0
	25%-solution sodium chloride	40.0
	sweetener	0.2
	buffer	0.5
	surface active agent	1.5
	sodium monofluorophosphate	1.0
	blended vegetable extracts	3.0
	aroma	1.5
	water - remainder	100.0

Example 5:	activated carbon as disclosed	10.0
	glycerin	10.0
	sodium alginate	0.6
	sodium saccharin	0.2
	sodium bicarbonate	50.0
	refined sodium chloride	5.0
	tincture of rhatany	1.5
	echinacin	1.0
	oil of mint	1.5
	1,-dimethyl-7-isopropyl azulene	0.1
	oil of sage	0.2
	preservative	0.1
	surface active compound	1.5
	buffer and water - remainder	100.0

Example 6:	activated carbon as disclosed	15.0
	glycerin	10.0
	70%-solution sorbitol	10.0
	sodium carboxymethyl cellulose	1.0
	silica thickening compound	1.0
	sodium saccharin	0.2
	sodium monofluorophosphate	2.0

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(cont...example 6

insoluble sodium metaphosphate	5.0
surface active compound	1.5
sodium bicarbonate	40.0
blended vegetable extracts	3.5
azulene	0.1
preservative	0.1
aroma	0.7
buffer and water - remainder	100.0

Example 7: activated carbon as disclosed	5.0
glycerin	20.0
binder	2.0
blended synthetic silicas	21.0
sweetener	0.2
buffer	1.0
surface active compound	1.5
preservative	0.1
aroma	1.0
water - remainder	100.0

Example 8:	activated carbon as disclosed	20.0
	70%-solution sorbitol	40.0
	propylene glycol	5.0
	sodium carboxymethyl cellulose	0.5
	blended synthetic silicas	10.0
	sodium bicarbonate	10.0
	alkyldimethylbenzyl ammonium saccharinate	0.1
	1-alpha-bisabolol	0.3
	blended aromas	1.0
	30%-solution surface active compound	3.0
	azulene	0.1
	quaternary ammonium compound	0.5
	neutralizer and water - remainder	100.0
Example 9:	activated carbon as disclosed	10.0
	70% solution sorbitol	50.0
	hydroxymethyl cellulose	0.7
	blended synthetic silicas	16.0
	sodium saccharin	0.2
	30% solution lauroyl sarcosinate	3.5
	buffer	8.8
	cetyl pyridinium chloride	0.2
	aqueous extract of tormentil	1.5
	aroma	1.0
	water - remainder	100.0

Composition - paste -----	% by weight -----
Example 10: activated carbon as disclosed	20.0
glycerin	30.0
dense precipitated calcium carbonate	5.0
anhydrous dicalcium sulphate	10.0
binder	1.5
silica thickening compound	2.0
surface active compound	1.0
sweetener	0.2
aroma	1.0
preservative	0.1
water - remainder	100.0

Example 11: activated carbon as disclosed	25.0
glycerin	10.0
70%-solution sorbitol	20.0
viscarin	1.0
dicalcium phosphate	10.0
tricalcium phosphate	2.0
dense precipitated calcium carbonate	2.0
sodium saccharinate	0.2

(over

(cont...example 11

sodium lauryl sulphate	0.8
N-lauroyl sarcosinate	0.3
germicidal	0.3
hydrated alumina	1.0
preservative	0.5
blended vegetable extracts	4.0
sodium bicarbonate	5.0
aroma	1.0
water - remainder	100.0

Example 12: activated carbon as disclosed	10.0
70%-solution sorbitol	20.0
satiagum	1.5
blended synthetic silicas	15.0
dense precipitated calcium carbonate	10.0
surface active agent	2.0
preservative	0.1
sweetener	0.2
blended aromas	1.0
water - remainder	100.0



The preparation of 100 kg of a gel according to the invention will now be described, by way strictly of example.

05 First, 5.0 kg glycerin, 38.0 kg sorbitol (in a 70% solution) and 4.5 kg sodium carboxymethyl cellulose are put into a suitable mixer and agitated until a uniform dispersion is achieved, following which, 5.0 kg activated carbon is admixed slowly and the mixing action continued to the point of obtaining a  
10 uniform blend. A solution of 18.7 kg deionized water with 0.2 kg sodium saccharinate and 0.9 kg buffer is then added, and the temperature of the blend raised slowly to 55...60 °C, with agitation continuing to the end of rendering the system  
15 homogeneous.

On reaching 55...60 °C and with the agitation still continuing, the next steps are to add 5.0 kg sodium chloride, step up the agitation to a vigorous pitch for some 15 minutes, and then add 13.0 kg blended  
20 synthetic amorphous silicas, agitating throughout. At this point, the blend is placed under vacuum conditions and deaerated.

Having restored ambient pressure, 1.5 kg blended surface active components are admixed to a uniform  
25 consistency, avoiding any entrainment of air, and

the mixture is returned to the vacuum and cooled, to 20...25 °C. Still under vacuum, 2.0 kg blended aqueous vegetable extracts are admixed to a uniform consistency, followed by 2.0 kg deionized water containing 0.2 kg of germicidal compound; mixing continues slowly until uniformity is achieved. Finally, 0.4 kg blended oleic vegetable extracts, 0.5 kg 1-alpha-bisabolol, 0.1 kg 1,4-dimethyl-7-isopropyl-azulene and 2.0 kg blended aromas are admixed in sequence, each to the point where a uniform consistency is achieved, maintaining the temperature and negative pressure conditions. The mixing action continues to be accompanied by deaeration until the product is free of lumps and air bubbles.

The gel thus obtained can then be packaged in the usual containers by conventional techniques. Naturally, the preferred compositions and method described in the foregoing specification are of a representative character and imply no limitation; in practice, use might be made of other equivalent chemical and methodological expedients without prejudice to the spirit of the appended claims.

## Claims

- 1) Compositions for tooth pastes and gels, characterized in that the cleansing-and-polishing, adsorbent-and-bleaching constituent consists exclusively or principally in orally innocuous activated carbon of fine granulometry and generous developable surface area.
- 2) Paste and gel compositions as in claim 1, wherein the activated carbon is of vegetable origin.
- 3) Paste and gel compositions as in claim 1, wherein the activated carbon content reflects between 0.5% and 50% by weight of the overall formulation.
- 4) Paste and gel compositions as in claim 1, wherein the average diameter of the activated carbon particles is between 2 and 20  $\mu$ m.

- 5) Paste and gel compositions as in claim 4, wherein the developable surface area of the activated carbon particles is not less than 300 m<sup>2</sup>/g.
- 6) Paste and gel compositions as in claim 1, wherein the activated carbon is of vegetable origin, consisting in particles of 5...15 m average diameter and 600...800 m<sup>2</sup>/g developable surface area, and reflects between 5% and 50% of the overall formulation.
- 7) Paste and gel compositions as in claim 1 or 6, comprising a quantity of saline electrolytes reflecting between 2% and 60% of the overall formulation.
- 8) Paste and gel compositions as in claim 7, wherein the saline electrolytes are sodium chloride and sodium bicarbonate.
- 9) Paste and gel compositions as in claim 1 or 6, comprising a quantity of substances producing a prophylactic or therapeutic action, that reflects between 0.01% and 5% of the overall formulation.

- 10) Paste and gel compositions as in claim 1 or 6, comprising at least one thickening agent.
- 11) Paste and gel compositions as in claim 10, wherein the thickening agent is a xerogel of silica or pyrogenic silica.
- 12) A method of preparing tooth paste/gel compositions as in claim 1 or 6, comprising the initial step, effected at ambient temperature, of obtaining a uniform dispersion of activated carbon in a blend of polyglycol vehicles and admixing a binder, or, of obtaining a dispersion of the binding agent in polyglycol vehicles, blended or otherwise, and . admixing the activated carbon.
- 13) Tooth paste and gel compositions and a method for their preparation as in preceding claims and in the foregoing specification, and as intended for the stated objects.
- 14) A composition for tooth pastes and gels substantially as described herein.
- 15) A method of preparing tooth paste/gel compositions substantially as described herein.